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TECHNICAL REPORT

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**DEVELOPMENT COMPOUNDING AND
EVALUATION OF PHOSPHAZENE RUBBER
FOR HELICOPTER SEAL APPLICATIONS**

by

Angus Wilson

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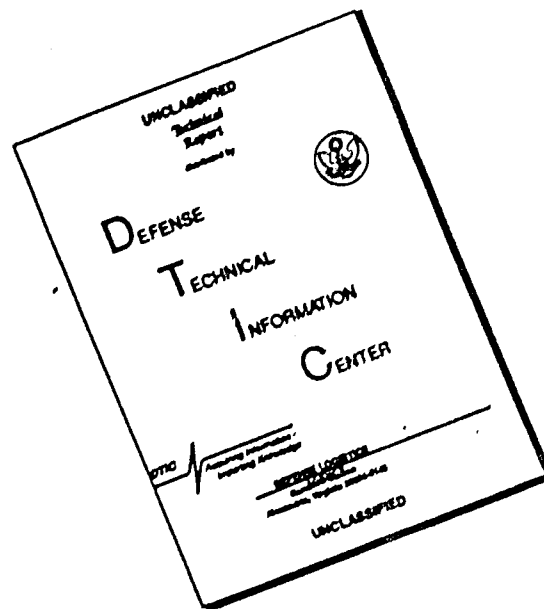
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A polyphosphazene copolymer, $\text{P}(\text{OCH}_2\text{CF}_3)_2 - \text{NP}(\text{OCH}_2\text{C}_6\text{F}_5\text{CF}_2\text{H})_2$ sub x was compounded with a variety of black and non-black reinforcing fillers, curing agents and other rubber additives. Optimum properties were obtained using silicas, silane treated clays, or combinations of these, in conjunction with peroxide curing agents. Tensile strengths of 116 Kg/cm (1650 psi) were achieved and lip seals were molded and gave evidence of potential use. The cured rubber was flexible to -54°C and showed good resistance to temperatures up to 150°C .		

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DEVELOPMENT COMPOUNDING AND EVALUATION
OF PHOSPHAZENE RUBBER FOR HELICOPTER SEAL APPLICATIONS

by

ANGUS F. WILSON

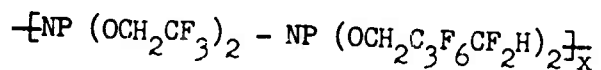
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FOREWORD

This report covers the results obtained from the development compounding and testing of a polyphosphazene rubber copolymer with the structure:



Objective of the work was the attainment of optimum strength, and the evaluation of physical properties and heat resistance with a view toward possible use of the material in helicopter seal applications.

The base rubber was supplied by Horizons, Inc., a contractor of the Army Materials and Mechanics Research Center (AMMRC). Work was carried out for the Organic Materials Laboratory of AMMRC under a customer order, PRON. NO. AW-1-Y1-1735-01-AW-BG. Funds for the project were furnished by AVSCOM under Project 1716042.

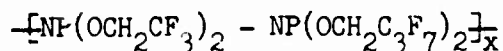
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Development Compounding of Phosphazene Rubber for
Helicopter Seal Applications

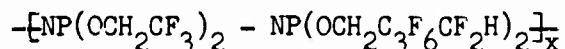
I. Introduction

Phosphazene rubber (or phosphonitrilic fluoro-elastomer) was developed under an Army Materials and Mechanics Research Center (AMMRC), Watertown, Massachusetts contract with Horizons, Inc., Cleveland, Ohio. The original work was directed toward the development of a fuel and oil resistant rubber that would remain flexible at low temperatures. Under the program several polymers were made and one of them, a copolymer with the structure:



underwent development compounding and testing at NLABS from 1968 to 1970. The data obtained showed the material to be a rubber with excellent fuel resistance, and with flexibility down to approximately -55°C , although the physical properties were poor, i.e. tensile strength below 61.2 Kg/cm^2 * (870 psi).^{1,2,3}

In 1972 AMMRC expanded the program with Horizons, Inc. to include the development of candidate phosphazene rubber compounds for evaluation as seal material for helicopter transmission shafts.⁴ The polymer chosen for this was a copolymer with the structure:



and the target properties sought were a Shore A durometer of 75 to 90, minimum tensile strength of 105.5 Kg/cm^2 (1500 psi), and minimum ultimate elongation of 175%. After heat aging 70 hours at 150°C the allowable maximum changes were to be +10 points durometer, -25% tensile strength, -30% ultimate elongation.

As part of the helicopter seal program AMMRC transferred funds to NLABS for development compounding to attain the target properties, and for evaluation of the materials produced. Compounding was also carried out by Horizons, Inc., suppliers of the polymer, and later in the program by personnel at the Mobility Equipment Research and Development Center at Fort Belvoir, Virginia.

*Tensile strengths are expressed as Kg/cm^2 in accordance with the ASTM method used in their measurement. If desired, the data can be converted from Kg/cm^2 to pascal by multiplying the given value by 9.806×10^4 .

II. Materials

Five batches of copolymer $\text{[NP(OCH}_2\text{CF}_3)_2 - \text{NP(OCH}_2\text{C}_3\text{F}_6\text{CF}_2\text{H)}_2\text{]}_x$ were received from April through November, 1972 and were identified as follows:

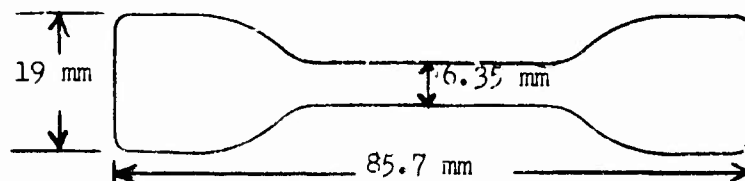
<u>Batch</u>	<u>Amount, grams</u>	<u>Viscosity, $[\eta]$, acetone</u>
1785-47A	246	1.85
1829-20	206	2.41
1869-34	450	1.8
1785-47A(2)	450	1.85
1931-07	910	1.71

The identity and source of other materials used in the compounding studies are given in the Appendix.

Tests on the raw polymer showed it to be soluble in acetone and methyl alcohol, and insoluble in benzene and 70/30 isooctane/toluene by volume.

III. Compounding, Curing, and Testing Procedures

Generally, batches using 5 grams of rubber were prepared. Ingredients were weighed on an analytical balance and compounded on a rubber mill with 25-mm (1-in.) diameter rolls, turning at a 10/7.5 speed ratio. After mixing, the rubber compound was removed from the 25-mm mill and sheeted to the desired thickness on a 76-mm (3-in.) mill. It was then molded and cured using a 1.9-mm (0.075-in.) thick mold of the following configuration and dimensions:



The use of teflon sheeting on either side of the mold cavity, required when molding previous phosphazene rubbers was unnecessary with this copolymer, and a light silicone spray coating was found sufficient to provide release.

Oven post curing, where used, was done in a circulating air oven of the type specified in ASTM Method D573-67.

Tensile and elongation testing was done according to ASTM Method D412-68, except that a non-standard test specimen (shaped to the mold dimensions shown above) was used.

Shore A durometer values were obtained according to ASTM Method D-2240-68. The tensile test specimen was used for this test and values were obtained on the tab ends of doubled specimens, backed up by another rubber material of similar durometer to provide sufficient thickness.

Low temperature flexibility (Gehman torsional stiffness) values were obtained according to D-1053-65.

IV. Results and Discussion

A. Preliminary Compounding

In order to compare the new material with the rubber previously evaluated, a sample of batch 1785-47-A, was compounded in a recipe which with the previous material had given a tensile strength of 61.2 Kg/cm^2 (870 psi) - the highest obtained. The recipe in parts by weight was as follows:

Polymer	100
Hisil 233	15
Magnesium oxide	5
Dicup 40C	4

The first three ingredients were mill mixed and bin-aged one week at room temperature prior to milling in the Dicup 40C. The specimen was then press-cured 1 hour at 143°C ; press-cooled; and oven-post-cured 1 hour at 93°C , 1 hour at 104°C , 1 hour at 121°C and 16 hours at 141°C ; and gave the following results:

Tensile strength	23.2 Kg/cm^2 (330 psi)
Ult. Elongation	200%
Shore A durometer	45

The compound was repeated but without bin-aging, and the material was mill mixed and press cured the same day, with the following results:

Tensile strength	44 Kg/cm^2 (625 psi)
Ult. Elongation	200%
Shore A durometer	45

Although part of this higher tensile result could be attributed to experimental difference, it appeared that bin-aging had no beneficial effect on the tensile strength of this compound.

Essentially the same compound was again mixed, except that 1.5 parts of stearic acid was added as a lubricant, and to remedy a tendency of the compound to stick to the small mill rolls during mixing. The addition, while resulting in a smoother sheet off the mill, did little to relieve the tendency to stick, and the cured material had many small surface bubbles. This batch also was mill mixed and press-cured the same day, and gave these results:

Tensile strength	42.2 Kg/cm ² (600 psi)
Ult. Elongation	250%
Shore A durometer	46

Since the recipe used above was a basic and simple formula, compounds were mixed eliminating various ingredients to determine their effect on physical properties. A compound was mixed containing only the polymer and dicumyl peroxide as follows:

Polymer	100
Dicup 40C	4
Press cured	60 min/127°C

Oven cured, for: 1 Hr/93°C, 1 Hr/104°C, 1 Hr/121°C, and then 16 Hr/141°C

This gave a tensile strength of less than 0.7 Kg/cm²(10 psi), comparable to that of a specimen of press-molded gum rubber.

To test the effect of magnesium oxide, the compound was again mixed, this time with the addition of 5 parts of the oxide, and press-cured and oven cured as before. The results this time were 17.6 Kg/cm²(250 psi) tensile strength, and 330% ultimate elongation. Thus, this initial series of compounds shows that dicumyl peroxide alone causes little, if any, effective curing of the phosphazene copolymer. Addition of magnesium oxide to the peroxide causes a marked improvement in the tensile strength, and the addition of a silica filler to these two ingredients increases the tensile again, by a factor of 2.5.

B. Fractionation

To check the possibility that the tensile strength of the rubber could be improved by removing its lower molecular portion, a small quantity of the raw gum was fractionated by

coagulation from an alcohol solution: 25 grams of raw polymer were dissolved in 200 ml. of reagent methanol, and 300 ml. of benzene was added dropwise, with stirring, over a period of $1\frac{1}{2}$ hours. The coagulated polymer was allowed to settle out and the liquid decanted. The polymer was air-dried at room temperature for 18 hours, and at 85° to 88°C for one hour. The yield was 80%, i.e. 20 grams, and the material was less tacky, and appeared somewhat tougher, than the parent polymer. The decanted liquid was evaporated at room temperature and the residue dried for one hour at 93°C . This material was flowable, soft and very tacky, with apparent low viscosity.

The coagulated rubber portion was cured according to the following recipes, with the following results:

Fractionated Polymer	100	100	100
Hisil 233	15	25	15
Magnesium oxide	5	5	5
Dicup 40C	4	2.5	2.5
Press cure Min/ $^{\circ}\text{C}$	60/143	60/143	60/143
Oven cure Hrs/ $^{\circ}\text{C}$	1/95	1/93	1/93
	1/104	1/104	1/104
	1/121	1/121	1/121
	16/141	16/141	16/141
Tensile, Kg/cm ² (psi)	47.1(670)	47.1(670)	40.8(580)
Ult. Elong., %	180	110	180
Shore A duro.	45	67	46

The data, when compared with that obtained on whole polymer, compounded in similar recipes (see above), shows no real superiority for the fractionated material, and would indicate that the presence of low viscosity material was not, or at least was not alone, responsible for the low strength values obtained on this copolymer.

C. Heat Treatment

The effect of heat applied during various phases of compounding was investigated using the following recipe:

Polymer 1785-47A	100
Meglite D	5
HsLL 235	18
A 172*	1
Disup 400	4

The compound was press-cured 1.3 hours at 143°C and oven cured 45 minutes at 93°C, 45 at 104°C, 45 at 121°C, and 16 hours at 143°C, and gave the following values:

Tensile strength	38.7 Kg/cm ² (550 psi)
Ult. Elongation	280%
Shore A durometer	50

The compound was repeated and all the ingredients except the dicumyl peroxide were milled together and oven heated for 16 hours at 100°C. It was then returned to the mill for addition of the peroxide and was cured as above, with these results:

Tensile strength	56.3 Kg/cm ² (800 psi)
Ult. Elongation	260%
Shore A durometer	51

The original compound was again repeated but no oven cure was used, and these values were obtained:

Tensile strength	66.8 Kg/cm ² (950 psi)
Ult. Elongation	280%
Shore A durometer	43

Another mix of the compound was made, in which all ingredients except the peroxide were mill mixed and oven heated for 16 hours at 100°C, prior to final milling, and which was not oven-postcured gave the following:

Tensile strength	70.3 Kg/cm ² (1000 psi)
Ult. Elongation	260%
Shore A durometer	40

The series shows that mill mixing and oven heating of ingredients before final mixing and curing improves physical properties, and that elimination of oven-postcure also results in improved properties. However, the combination of preheating and oven cure elimination gave only slightly higher values

*A 172 is an organo silane (see Appendix), one of a class of materials currently finding use in rubber compounding to promote interaction between rubber and non-black fillers (5).

than elimination of oven cure alone, and the difference, in fact, can be attributed to normal test variation. The results strongly indicate that lengthy oven-postcuring is unnecessary with the dicumyl peroxide cure system.

There was a possibility that the heat resistance of the cured rubber could be improved by heating the raw rubber prior to compounding, on the hypothesis that such treatment would eliminate those portions of polymer most heat susceptible and leave a residue of heat resistant material. Accordingly a 15-gram sample of raw rubber was heated for 96 hours at 149°C. The material lost 0.5% of its weight and became dark, transparent, and flowed out flat and level. It was compounded using the recipe given above and gave a soft putty-like mass, which when press-cured for 60 minutes at 143°C gave a tensile of less than 0.7 Kg/cm² (10 psi).

A second sample of raw rubber was mill mixed with the Hisil 233 (from the above recipe) and then heated for 96 hours at 149°C. This material lost 1% of its weight but did not darken to the extent that the raw rubber had, and did not flow, or lose its shape. It was compounded with the remaining recipe ingredients and press-cured, and gave a tensile strength of less than 0.7 Kg/cm² (10 psi).

These mixes clearly show that the heating at 149°C in air results in severe degradation of the base polymer, probably due to depolymerization. Allcock, Kugel and Valan⁶ had demonstrated that methoxy, ethoxy, trifluoroethoxy and phenoxy derivatives of $(\text{NPCl}_2)_n$ suffered depolymerization below 200°C, and that solution viscosity measurements showed a viscosity decrease after heating 8 hours at 150°C. Further, their work showed retention of form and integrity when treated in a vacuum at 200°C, but depolymerization in nitrogen at 150°C and 200°C.

D. Solution Mixing

To check the efficiency of the milling procedure, especially in the mixing of the filler into the base rubber, a compound was made by dissolving the rubber and then adding the filler; as follows:

Twelve grams of polymer 1785-47A were dissolved in 100 ml of methanol, and 2.16 gms of Hisil 233 (equivalent to 18 parts per hundred parts of rubber) were stirred into the solution. The mixture was allowed to stand for three

days and then poured into an evaporating dish with three methanol rinsings of 15 ml each. The methanol was allowed to evaporate away at room temperature for one day. The polymer/filler residue was dried for 2 hours at 80°C, mill mixed with magnesium oxide and dicumyl peroxide, and press and oven cured. The results obtained were:

Tensile strength	72.1 Kg/cm ² (1025 psi)
Ult. Elongation	310%
Shore A durometer	36

These results were comparable to the results obtained on mill mixed stocks discussed in the previous section and showed mixing on the 1" mill to be an efficient mixing procedure.

E. General Compounding

Two batches of polymers (1785-47A and 1829-20) were used to make a series of compounds in which both rubbers were formulated in the same recipes. This, in effect, gave a duplicate test of the individual recipes and provided an opportunity to observe any trend toward a difference in the properties of the two base rubbers. The compounds formulated and the results obtained, listed in Table I, show that in most instances there was no significant difference between the two batches of polymer. The sole exception was the pair 47-A-10 and 20-10 made with filler of silica and carbon black, with which the 1829-20 polymer gave a tensile strength of 70 Kg/cm² (995 psi), compared to 14 Kg/cm² (200 psi) for the 1785 - 47A polymer.

Double batches of these two compounds were prepared, split in half, and press-cured. One specimen of each was oven-postcured and the other was not. Compound 1829-20 gave a tensile of 67 Kg/cm² (955 psi) with oven cure and 56 Kg/cm² (795 psi) without; 1785-47A gave 46 Kg/cm² (655 psi) with oven cure and 39 Kg/cm² (555 psi) without. Although the 1829-20 polymer again gave the higher strength results, the difference was not as great as with the previous mix. Another mix of the compounds was prepared, this time with results of 67 and 26 Kg/cm² (955 and 355 psi) respectively. The two compounds were then mixed with a,a'-bis(t-butyl peroxy) diisopropylbenzene (Vulcup 40KE) substituted for the dicumyl peroxide with similar results of 65 and 17 Kg/cm² (925 and 245 psi). Thus the results show the tensile values obtained with polymer 1829-20 to be consistent, while those with polymer 1785-47A to be erratic, indicating a sensitivity of this material to the silica/carbon black filler in conjunction with peroxide cures. The basic reason for this is not apparent.

Otherwise, a review of the results contained in Table I (see Appendix B) shows that the highest strength values were obtained with the silica filler, dicumyl peroxide cure (54.8 Kg/cm^2) (780 psi) and with a HAF black filler, dicumyl peroxide cure (59 Kg/cm^2) (840 psi), values still short of the target value of 105 Kg/cm^2 (1495 psi).

The use of phenol-formaldehyde resins (Amberol and Durez) in conjunction with either peroxide or amine cures gave poor tensile properties. Likewise, the use of the Diaks, developed for curing fluoroelastomers, resulted in complete degradation of polymer strength properties. This could have been due either to the effect of materials themselves, the higher temperatures required for their activation, or a combination of these factors.

In order to conserve polymer, since the amounts available were relatively small, the practice of making a group of compounds from two different batches of polymer was discontinued. Instead a series of compounds was made to evaluate the effects of various curing agents, fillers, coupling agents, and other additives. The recipes used and the physical properties obtained are given in Table II.

Analysis of the data allows several conclusions to be drawn.

The use of a base, such as TETA or 2,6-diaminopyridine in conjunction with dicumyl peroxide (47-A-12, 57-A-16, 34-K) resulted in tensile reductions of up to 50%.

A silica/HAF black filler combination (47-A-17) gave mediocre physical properties.

The blending of polyphosphazene polymer with ethylene propylene diene rubber (20-11, 20-12) gave tensile properties inferior to those of straight polyphosphazene.

The use of dibasic lead phosphite (Dyphos) in a silica filled and dicumyl peroxide cured stock (20-13, 20-14) slightly improved tensile properties.

A group of compounds made to investigate dicumyl peroxide levels and cure times (34-A thru 34-H) showed that 2 parts of Dicap 40C gave tensile properties equivalent to, or slightly higher than, those obtained with 4 parts, and that better properties were attained without oven postcure than with it. This is in agreement with results contained in Table I, previously discussed.

Variations in peroxide levels in conjunction with the use of dibasic lead phosphite, in compounds subjected to oven heating prior to peroxide addition (34-I, 34-J, 34-I Rpt and 34-J Rpt) showed no difference between 2 and 4 parts of Dicap 400, and no tensile improvement resulting from oven preheating. However, the dibasic lead phosphite, sold commercially as a light and heat stabilizer for chlorinated rubbers and vinyls, may diminish the effects normally resulting from heat.

Compounding without any filler other than 20 parts of dibasic lead phosphite (34-J) gave a very low tensile.

Compounding with a high level of silica (50 parts) with and without the coagent Chemlink 30 (34-M, 34-N) gave a stiff material incapable of being bent without breaking.

Vulcup 40KE (40% a-a'bis(t-butyl peroxy) diisopropylbenzene on clay) in conjunction with varying amounts of silica (34-O, 34-P, 34-Q) gave tensiles up to 91.4 Kg/cm^2 (1300 psi), but in the presence of 2,6-diaminopyridine (34-R) gave poor results. The use of a coagent, Chemlink 30, with the Vulcup (34-S) did not improve the strength.

The use of a clay filler, such as Burgess OFE (34-T through 34-W) resulted in poor to mediocre cures. The use of a silane treated clay, Burgess KE, alone or in combination with a silica (34-AN, 34-AO) showed substantial improvement, 56 to 74 Kg/cm^2 (800 to 1055 psi) compared to 15 to 44 Kg/cm^2 (215 to 625 psi).

A series of compounds using a variety of clay, black and silica fillers (34-X, 34-Y, 34-Z, 34-AA through 34-AP, 34-AT through 34-AZ, and 34-BA through 34-BH), in combination with Vulcup curing agent ranged in tensile strength from 21 to 116 Kg/cm^2 (300 to 1650 psi). Those meeting the target tensile of 105.5 Kg/cm^2 (1500 psi) were compounds using a 18/40 combination of silica and silane treated clay (34-AC), a silane treated silica 34-AH, and combinations of silane treated clay and silane treated silica (34-AI, 34-AJ, 34-AK). In all of the batches additional silane was added to the fillers prior to milling. The highest tensile strength achieved was in compound 34-AI with a value of 116.0 Kg/cm^2 (1650 psi), evidently because of the combination of clay, silica, and silanes used in the filler system.

Three additional peroxides; 2,5-dimethyl-2,5-bis(t-butyl peroxy) hexane; 2-5-dimethyl-2,5-bis(t-butyl peroxy) hexyne-3; and di-t-butyl peroxide (34-AQ, 34-AR, 34-AS) all failed to achieve the tensile strength achieved with the Vulcup 40KE.

Two additional batches of raw rubber evaluated in Vulcup cured stocks using silica/clay filler combinations (07-A, 07-B, 47-A through 47-D) gave slightly lower tensile values than those obtained with the previously evaluated rubber (34-AI), i.e. 98.4 and 102.0 Kg/cm² (1400 & 1450 psi) compared with 116 Kg/cm² (1650 psi).

A series of compounds containing varying amounts of silane coupling agents in conjunction with a fumed silica filler (Aerosil 200) and cured with Vulcup (47-G through 47-V) showed no advantage over the base compound made without silane (47-F). Of the four silanes used: A151, vinyl-triethoxy silane; A171, vinyltrimethoxy silane; A172, vinyl-tris (beta-methoxyethoxy) silane; and A1100, gamma-aminopropyltriethoxy silane; 2 parts of A1100 gave the highest tensile value, 91.4 Kg/cm² (1300 psi); but this also was attained with no silane and the results indicate that no beneficial effect is obtained from addition of silane to this filler, in this polymer.

Use of alumina filler or alumina/silica blends (47-Alon, 47-Alonsil A, 47-Alonsil B) gave mediocre strength values, 81 Kg/cm² (1150 psi) or less.

The use of hydrophillic precipitated silicas (47-W through 47-AB), or naturally occurring amorphous silicas (47-AE through 47-AH) resulted in low strength values.

As previously discussed the highest tensile achieved in the Table II compounds was 116 Kg/cm² (1650 psi). However, Horizons, Inc., producers of the various batches of polymer had achieved tensile values in the 140 Kg/cm² (2000 psi) range, and with one compound had obtained 150.8 Kg/cm² (2145 psi). In order to evaluate the consistency of properties between different batches of raw rubber, Horizons' recipe was used with polymer 1785-47A (47-AC and 47-AD). Samples of the individual compounding ingredients were obtained from Horizons and used to mix the compound and, except for the peroxide, were either oven dried, milled together and bin aged; or vacuum oven dried, milled together and bin aged prior to peroxide addition and cure—the latter procedure being one followed by Horizons. The highest tensile obtained was 80.9 Kg/cm² (1150 psi). A similar series prepared

using Vulcup 40KE, instead of the Luperox 500 40KE provided by Horizons, gave a high tensile of 91.4 Kg/cm^2 (1300 psi). The difference of 70 Kg/cm^2 (1000 psi) between Horizons' and our compounding of the same recipe is strong indication of either basic strength differences in various batches of the polymer or strong sensitivity of the polymer to differences in compound mixing procedures.

The effects of various heat stabilizers- Dyphos (dibasic lead phosphite), Dythal XL (dibasic lead phtalate), and Leadstar (normal lead stearate) - in prevention of deterioration during cure were investigated in compounds 47XX0 through 47XX3 and 47ZZ0 through 47ZZ3. Dyphos slightly improved the tensile strength, Dythal XL slightly decreased it, and Leadstar resulted in approximately a 50% reduction. One part of SRF black was also evaluated (47XX4 and 47ZZ4) because work by J. A. Williams showed that a small amount of black acts as a heat stabilizer in silicone rubber. In these polyphosphazene rubbers, however, it had little effect.

In Table III are shown results obtained on a series of compounds made with polymer batch No. 1931-07. These include various black and non-black fillers, blends with another polymer, and the use of various compounding additives - all compounds being cured with Vulcup 40KE. The series again clearly demonstrates that little tensile strength can be developed without a reinforcing filler, and that the best tensile values could be achieved using the fumed silica or a silica/silane-treated-clay combination. The highest value obtained was 98.4 Kg/cm^2 (1400 psi), using such a combination. The results clearly demonstrated that carbon black fillers generally gave very poor results and in many cases prevented curing. The best tensile obtained with a black was 63.3 Kg/cm^2 (900 psi), and this was in a compound using a blend of polyphosphazene rubber and EPDM rubber (02-G). Blending of the two rubbers resulted in low tensile values, the above value being the highest obtained.

The series also showed that a satisfactory cure could be obtained by substituting zinc oxide for magnesium oxide, and that the use of ultra-fine talc particles as a filler produced stocks with moderate tensile strength; i.e. approximately 49 Kg/cm^2 (700 psi).

F. High Temperature Aging and Seal Testing

In order to evaluate the ability of the polyphosphazene rubber to meet the target requirements for resistance to heat aging, compound 34-AC (Table II) was mixed, press cured 20 minutes at 160°C and oven cured 18 hours at 100°C. Physical properties were measured before and after aging 70 hours at 149°C, with the following results:

	Original	Aged	Change	Target Change
Tensile, Kg/cm ²	112.5	91.4	-19%	-25% Max.
Elong., %	80	75	- 6%	-30% Max.
Shore A, Duro. Points	85	88	+ 3	+10 Max.

The tests showed good retention of properties, under these aging conditions, and as a result a 50-gram batch of the compound was milled without peroxide for fabrication into test seals. A small portion of the compound was sampled at NLABS, milled with the peroxide curing agent, cured and tested, with these results:

Tensile: 112.5 Kg/cm² (1600 psi)

Elongation: 90%

Shore A: 79

The remainder of the compound and curing agent was sent to Federal-Mogul, Inc. a contractor engaged in testing prototype helicopter seal materials supplied by Horizons, Inc. The curative was milled into the compound and seals were fabricated by Federal-Mogul for evaluation on their test equipment.

As reported by Federal-Mogul⁸, the test conditions were: MIL-L-7808F, 230°F, 5500 RPM, 20 hours on, 4 off, test to be terminated when measurable leakage (of the lubricant) occurred; with these results:

Seal 1: dry at 18.0 hours, leaked 16 grams after 42 hours.

Seal 2: dry at 17.5 hours, leaked 5.9 grams after 89.5 hours.

These values were intermediate between those obtained by Federal-Mogul on other polyphosphazene compounds.

G. Low Temperature Properties

A compound containing 15 parts of silica, 5 parts of magnesium oxide, and 4 parts of Dicap 40C (which, when press and oven cured, gave a tensile strength of 23.2 Kg/cm² and a Shore durometer value of 45) was tested for low

temperature stiffness according to ASTM Method D 1053-65, with the following results:

T_2	-47.5°C
T_5	-54.5
T_{10}	-57.5
T_{100}	-66.0

V. Summary and Conclusions

The copolymer, $[\text{NP}(\text{OCH}_2\text{CF}_3)_2 - \text{NP}(\text{OCH}_2\text{C}_3\text{F}_6\text{CF}_2\text{H})_2]_x$ is curable with various peroxides, and the best results were obtained with dicumyl peroxide (39.5 - 41.5% active supported calcium carbonate) and a *a-a'*-bis(*t*-butyl peroxy) diisopropylbenzene (39.5 - 41.5% active supported on silane treated clay).

The best reinforcing fillers for the copolymer were precipitated and fumed silicas, silane treated clays and combinations of these. Carbon blacks in general gave poor results and in some cases interfered with peroxide cures.

The cured rubber exhibited good aging resistance and low temperature flexibility down to -54°C.

The target properties were exceeded, and tensile strengths of 116 Kg/cm² (1650 psi) were achieved, and with the use of proper heat stabilizers higher strengths might be obtained.

VI. Future Work

Work will continue at NLABS on compounding of these and other polyphosphazene rubbers, under an AMMRC customer order. Emphasis will be placed on achieving optimum physical properties and on developing compounds suitable for specific fabrication techniques, including dipping and coating.

VII. References

- (1) Wilson, A. Initial Compounding Studies of Polyphosphazene Rubber, TR 70-10-CE, (C&PLSEL-68) US Army Natick Labs., August 1969.
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- (3) Wilson, A. Polyphosphazene Rubber; Horizons, Inc., No. 1487-12. Memo Report, US Army Natick Labs., April 1970.
- (4) Reynard, R. A., Sicka, R. W., Vicic, J. C., Rose, S. H., Development of Thermally Stable Poly(Fluoroalkoxyphosphazene) for the UH-1 Helicopter, Final Report - Contract DAAG 46-72-C-0073, September 1973
- (5) Ziemianski, L. P., Pagano, C. A. and Ranney, M. W. Silanes in Elastomers: New Route to High Performance Pigmented Products. Rubber World, October 1970.
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- (7) Williams, John A. Carbon Black as a Heat Stabilizer in Silicone Rubber Vulcanizates, SWERR-TR-72-28, Weapons Laboratory at Rock Island, US Army Weapons Command, April 1972.
- (8) Inter-Office Memorandum, R. Hinderer to J. Born, Federal-Mogul, May 24, 1973.

Appendix A
Identification of Compounding Ingredients

<u>Material</u>	<u>Composition</u>	<u>Source</u>
Aerosil 200	Hydrophillic fumed silica	Degussa, Inc.
Aerosil 300	Hydrophillic fumed silica	Degussa, Inc.
Aerosil R972	Fumed silica treated with dimethyldichloro silane	Degussa, Inc.
Agarite Resin D	Polymerized 1,2-dihydro-2,2,4-trimethylquinoline	R.T. Vanderbilt Co., Inc.
Alon	Fumed Alumina	Cabot Corporation
Amberol ST-137X	Phenol-formaldehyde resin	Rohm & Haas Co.
Burgess KE	Silane treated aluminum silicate	Burgess Pigment Co.
Burgess OPE	Aluminum silicate	Burgess Pigment Co.
Cebbowax 4000	Polyethylene-glycol	Union Carbide, Corp.
Chemlink 30	Trimethylol Propane Trimethacrylate	Ware Chemical Corp.
Diak 5	Treated hydroquinone	E.I. duPont deNemours & Co.
Diak 6	Proprietary, fluorocarbon curing agent	E.I. duPont deNemours & Co.
Dicup 40C	40% dicumyl peroxide on calcium carbonate	Hercules, Inc.
Durez 12687	Phenol-formaldehyde resin	Hooker Chemical Corp.
Dyphos	Dibasic lead phosphite	National Lead Co.
Dythal SL	Dibasic lead phthalate	National Lead Co.
Elastomag 170	Magnesium oxide	Morton Chemical Co.
Hisil 233	Precipitated hydrated silica	PPG Industries
Imsil A10	Amorphous silica	Illinois Minerals Co.
Imsil A15	Amorphous silica	Illinois Minerals Co.
Leadstar	Normal lead stearate	National Lead Co.
Luperco 101 XL	2,5-Dimethyl-2,5-bis(t-butyl peroxy) hexane	Penwalt, Lucidol Div.
Luperco 130 XL	2,5-dimethyl-2,5-bis(t-butyl peroxy) hexyne-3	Penwalt, Lucidol Div.
Maglite D	Magnesium oxide	Merck & Co., Inc.
Mistron Vapor	Magnesium silicate	Cyprus Mines, United Sierra Div.
Nordel 1040	Ethylene-propylene-diene terpolymer	E.I. duPont deNemours & Co.
Nulok 321 SP	Silane treated aluminum silicate	J.M. Huber Corp.
QUSO F22	Precipitated silica	Philadelphia Quartz Co.
QUSO G32	Precipitated silica	Philadelphia Quartz Co.
QUSO H40	Precipitated silica	Philadelphia Quartz Co.
QUSO WR82	Precipitated silica	Philadelphia Quartz Co.
Royalene 400	Ethylene-propylene-diene terpolymer	Uniroyal Chemical Div.
Silane A151	Vinyltriethoxy silane	Union Carbide Corp.
Silane A171	Vinyltrimethoxy silane	Union Carbide Corp.
Silane A172	Vinyl silane	Union Carbide Corp.
Silane A1100	Gamma-aminopropyltriethoxy silane	Union Carbide Corp.
Silancx 101	Fumed silica	Cabot Corp.
Silene EF	Precipitated hydrated calcium silicate	PPG Industries, Inc.
Vulcup 40KE	40% a-a'bis(t-butyl peroxy) di-isopropylbenzene on Burgess KE clay	Hercules, Inc.

Appendix B

Tables of Compounding Ingredients and Test Results

- Table I Duplicate Compounds of Base Polymers
1785-47A and 1829-20.
- Table II Compounds of Base Polymers 1785-47A,
1829-20, and 1869-34.
- Table III Compoun of Base Polymer 1931-07.

TABLE I

Duplicate Compounds of Base Polymers 1785-47A and 1829-20
(parts by weight, and test results)

Batch Number	47A1	20-1	47A2	20-2	X	47A3	20-3	47A4	20-4	47A5	20-5	47A6	20-6	47A7	20-7	47A8	20-8	47A9	20-9	47A10	20-10
Polymer 1785-47A	100		100		50	100		100		100		100		100		100		100		100	
Polymer 1829-20		100		100	50		100		100		100		100		100		100		100		100
Maglite D	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Zinc Oxide																					
CaOH																					
Hisil 233	15	15			7.5			15	15	15	15	15	15	15	15					10	10
HAF Black			15	15	7.5													25	25	15	15
MT Black						15	15									15	15				
Amberol ST 137X								8	8	8	8										
Durez 12687												8	8								
Dicup 40C	4	4	4	4	4	4	4	4	4									4	4	4	4
Diak 5																	0.8				
Diak 6																	1.1				
Triethylene tetramine										5	3	3	3	3	3						
press cure, min/°C	60/143									60/127	60/127					20/119	60/143				

TABLE I (Continued)

Duplicate Compounds of Base Polymers 1785-47A and 1829-20
(parts by weight, and test results)

Batch Number	47A1	20-1	47A2	20-2	X	47A2	20-3	47A4	20-4	47A5	20-5	47A6	20-6	47A7	20-7	47A8	20-8	47A9	20-9	47A10	20-10
Oven cure, hr/°C	.75/93	→	→	→	→	→	→	→	→	→	→	.75/104	→	.75/93	→	.75/93	→	.75/93	→	→	→
	.75/104	→	→	→	→	→	→	→	→	→	→	.75/121	→	.75/104	→	.75/113	→	.75/104	→	→	→
	.75/121	→	→	→	→	→	→	→	→	→	→	16/143	→	.75/121	→	.75/135	→	.75/121	→	→	→
	16/143	→	→	→	→	→	→	→	→	→	→			16/141	→	16/157	→	16/141	→	→	→
Tensile, Kg/cm ²	54.8	49.2	42.2	58.6	31.6	35.1	21.1	3.5	3.5	2.8	10.5	10.5	23.1	1.4	8.7	Broke	0	7.0	10.5	14.1	70.3
Ult. Elong, %	250	180	400	280	210	300	180	300	320	400	500	200	110	500	500	during	1100	320	400	330	280
Shore A Durometer	48	45	30	23	30	23	26	40	38	40	35	40	40	32	30	cure	23	42	43	44	42

TABLE II

Compounds of Base Polymers 1785-47A, 1829-20, and 1869-34
(parts by weight and test results)

Batch Number	47A12	47A16	47A17	20-11	20-12	20-13	20-14	34-A	34-B	34-C	34-D	34-E	34-F	34-G	34-H	34-I	34-J	Rpt. 34-I	Rpt. 34-J
Polymer 1785-47A	100	100	100*																
Polymer 1829-20				70*	100	100	100												
Polymer 1869-34								100	100	100	100	100	100	100	100	100	100	100*	100*
Nordel 1040				30*															
Maglite D	5	5	5*	5*	5	5	5	5	5	5	5	5	5	5	5	5	5	5*	5*
Dyphos							5									5	5	5*	5*
HAF Black			15*																
Hsil 233	18	18	10*	20*	18	18	18	18	18	18	18	18	18	18	18	18	18	18*	18*
Carbowax 4000	0.5																		
Silane A1100				1.1*	1	1.2	1.2												
Silane A172	1	1	1*													1	1	1*	1*
Triethylene tetramine		1																	
Dicup 400	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4	2
Press cure, min/°C	60/143									15/143	60/143	60/143	60/143	15/143	15/143	60/143	60/143	15/143	60/143

TABLE II (Continued)

Compounds of Base Polymers 1785-47A, 1829-20, and 1869-34
(parts by weight and test results)

Batch Number	47A12	47A16	47A17	20-11	20-12	20-13	20-14	34-A	34-B	34-C	34-D	34-E	34-F	34-G	34-H	34-I	34-J	Rpt. 34-I	Rpt. 34-J
Oven cure, Hr/°C			.5/93 .6/104 .6/121 16/143	→	→	→	→		.75/93 .75/104 .75/121 16/143	16/143	16/143		.75/93 .75/104 .75/121 16/143		16/143				
Special Treatment			*Milled and heated 16 Hrs. @ 100°C.	*Milled and heated 72 Hrs. @ 82°C.														*Milled and heated 66 Hrs. @ 93°C.	→
Tensile, Kg/cm ²	56.3	28.1	35.2	21.1	49.2	49.2	62.3	54.4	54.4	59.8	47.5	66.8	66.8	56.2	49.2	66.8	66.8	70.3	66.8
Ult. Elongation, %	310	350	300	150	200	200	180	170	150	130	140	120	100	110	120	190	200	180	180
Shore A, Durometer	36	39	42	53	42	45	45	53	54	48	53	49	55	50	52	42	42	41	41

TABLE II (Continued)

Compounds of Base Polymers 1785-474, 1829-20, and 1869-34
(parts by weight and test results)

Batch Number	34-Z	34-L	34-M	34-N	34-O	34-P	34-Q	34-R	34-S	34-T	34-U	34-V	34-W	34-X	34-Y	34-Z	34-AA	34-AB	34-AC	Rpt. 34AC
Polymer 1869-34	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Maglite D	5	5	5	5	5*	5*	5*	5*	5*	6*	5*	6*	5*	5*	5*	5*	5*	5*	5*	5*
Dyphos		20				5*	5*	5*	5*		5*		5*	5*	5*	5*	5*	5*	5*	5*
2,6 diaminopyridine	5			2				2												
Chemlink 30									1.5	1.5		1.5								
Hsil 233	18		50	50	20*		28*	30*	30*							20*				
Burgess OPE									100*	100*	100*	100*	100*							
Mistron Vapor														80*		35*				
Aerosil 200															30*		23*		18*	18*
Nulok 321-SP																		80*	40*	40*
Silane A172	1		1	1	1	1	1	1.5	1					1	1	1	1	1	1	1
Dicup 40C	4	4	4							7.5										
Vulcup 40 KE					4	4	4	5	5		4	7.5	7.5	6	4	5	4	5	5	5
Press cure, Min/°C	60/143									15/160			60/143					20/160		
Oven cure, Hr/°C	1.5/100				1/100					24/100			1/100			16/100	1/100	40/100		18/100

TABLE II (Continued)

Compounds of Base Polymers 1785-47A, 1829-20, and 1869-34
(parts by weight and test results)

Batch Number	24-K	24-L	24-M	24-N	24-O	24-P	24-Q	24-R	24-S	24-T	24-U	24-V	24-W	24-X	24-Y	24-Z	24-AA	24-AB	24-AC	Rep. 24AC
Special Treatment					*Oven dried @ 127°C					Milled & Bin Aged 24 Hrs.		Milled & Bin Aged 24 Hrs.								
Tensile, Kg/cm^2	35.2	17.6	Stiff	Stiff	80.9	91.4	91.4	28.1	84.4	44.3	17.6	15.6	19.7	42.2	91.4	84.4	91.4	87.9	105.5	112.5
Ult. Elongation, %	>500	300	Stiff & Frt-able	Stiff & Frt-able	200	150	160	320	150	120	150	180	150	120	180	110	160	80	90	80
Shore A. Durometer	40	25			43	72	60	45	72	62	>7	60	58	54	90	77	75	72	85	85

TABLE II (Continued)

Compounds of Base Polymers 1785-47A, 1829-20, and 1869-34
(parts by weight and test results)

Batch Number	34AD	34AE	34AF	34AG	34AH	34AI	34AJ	34AK	34AL	34AM	34AN	34AO	34AP	34AQ	34AR	34AS	34AT	34AU
Polymer 1869-34	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Maglite D	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*
Dyphos	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*
Burgess OPE		80*																
ISAF Black	25*		20*															
Nulok 321 SP																		
Burgess KE						40*	40*	20*	40*									
Aerosil 200			15*							25*		40*						
Aerosil 300														30*	30*	30*		
Aerosil R972				23*	28*	23*	30*	30*	23*			20*						
Silene EF													50*					
QUSO G32																	25*	25*
QUSO WR82																		
Carbowax 4000												0.5						

TABLE II (Continued)

Compounds of Base Polymers 1785-47A, 1820-20, and 1869-34
(parts by weight and test results)

Batch Number	34AD	34AE	34AF	34AG	34AH	Rdt. 34AH	34AI	34AJ	34AK	34AL	34AM	34AN	34AO	34AP	34AQ	34AR	34AS	34AT	34AU
Silane A172				1	1	1	1	1	1	1	1		1	1	1	1	1	1	1
Luperc 101AL															4				
Luperc 130AL																4			
Di - t-Butyl Peroxide																	3		
Vulcup 40KE	5	5	5	4	4	4	5	5	5	3	4	4	4	4				4	4
Press Cure, min/°C	20/160	→	→	60/143	→	→	20/160	→	→	25/171/120/160	→	→	→	→	→	→	60/143	20/160	→
Oven Cure, hr./°C	16/100	40/100	16/100	16/100	18/100		18/100	→	→		1/100	→	→	→	→	→	→	→	→
Special Treatment	*Oven dried @ 127°C	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→
Tensile, Kg/cm ²	28.1	21.1	38.7	87.9	98.4	105.5	116.0	112.5	105.5	94.9	84.4	56.2	73.8	70.3	49.2	42.2	45.7	63.3	66.8
Ult. Elongation, %	300	200	200	180	170	180	80	75	90	80	140	100	90	100	110	100	180	120	200
Shore A Durometer	45	58	78	56	70	64	80	85	79	75	84	69	84	76	88	88	83	53	50

TABLE II, Cont.

Batch No.	24AV	24AW	24AX	24AY	24AZ	24BA	24BB	24BC	24BD	24BE	24BF	24BG	24BH	07A	07B	47A	47B	47C	47D	47E
Polymer 1869-34	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100					
Polymer 1931-07														100	100					
Polymer 1785-47A(2)																				
Kaglite D	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	100	100	100	100	100
Dyphos	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	6*
Quso 63C	15*			40*																5*
Quso MR 82		15*	15*		40*															
Rulok 321 SP			40*			80*	50*	35*	15*	10*	50*	40*	40*	40*	40*	40*	40*	40*	40*	40*
Aerosil R972						7.5*	15*	25*	35*	23*	20*	23*	23*		23*					
Aerosil 200														18*		18*		18*	18*	20*
Silane A172	1	1	1	1.5	1.5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Silane A1100																				1.5
Chemlink 30																				
Vulcup 40KE	4	4	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	4
Press Cure, min/°C	20/160																			
Oven Cure, hr/°C	1/100																			
Special Treatment	*Oven dried @127°C																			
Tensile, Kg/cm ²	56.2	60.0	91.4	38.7	56.2	98.4	91.4	98.4	94.9	87.9	103.0	91.4	91.4	73.8	98.4	102.0	94.9	91.4	63.3	84.4
Ult. Elongation, %	160	250	100	110	130	80	90	90	90	110	110	100	90	70	90	90	80	75	110	70
Shore A durometer	32	28	60	75	70	70	72	75	80	64	68	59	71	70	60	78	75	83	73	83

TABLE II, Continued

Batch Number	47E	47G	47H	47I	47J	47K	47L	47M	47N	47O	47P	47Q	47R	47S	47T	47U	47V	47 ALON [®]	47-Alonsil	47-Alonsil A	47Alonsil B
Polymer 1785-47A	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Maglite D	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*
Alonsil 20C	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*	25*
Alon																					
Silane A151	1	1.5	2	2.5																	
Silane A171																					
Silane A172																					
Silane A100																					
Vulcup 40RE	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Press Cure, min/°C	20/160																				
Oven Cure, hr/°C	1/121																				
Special Treatment	*Oven dried @127°C																				
Tensile, Kg/cm ²	102.0	180.5	70.3	77.3	49.4	70.3	84.4	84.4	56.2	70.3	80.9	77.3	66.8	73.8	87.9	91.4	77.3	49.2	67	80.9	77.3
Ult. Elongation, %	120	120	150	175	120	110	120	140	100	140	220	260	270	80	120	110	100	180	140	110	90
Shore A, durometer	87	82	82	81	83	83	81	82	79	77	76	77	73	77	73	77	69	43	54	75	65

TABLE II. Cont.

Batch No.		47W		47X		47Y		47AA		47AB		47AC		47AD		47AD		47AE		47AF		47AG		47AH	
Polymer	1785-47A	100	100	100	100	100	100	100	100	100	100	100*	100*	100	100*	100*	100	100	100	100	100	100	100	100	
Maglite D	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*	6*	6*	6*	6*	6*	6*	5*	5*	5*	5*	5*	5*		
Elastomag 170																									
Dyphos																									
QUSO H40	18*		25*		32*																				
QUSO F22				25*		32*						30*	30*	30*	30*	30*	30*								
Silanox 101																									
Imsil A10																									
Imsil A15																									
Vulcup 40 KE	5	5	5	5	5	5	5	5	5	6	6	6	6	6	6	6	6	5	5	5	5	5	5		
Luperox 500 40 KE																									
Press cure, Min. / \bar{c}	20/160																	20/160							
Oven cure, Hr. / \bar{c}	1/121																	1/100							
Special Treatment	*oven dried @ 127°C																	*Oven dried @ 127°C							

TABLE II, Cont.

Batch No.	47XX0	47XX1	47XX2	47XX3	47XX4	47Z20	47Z21	47Z22	47Z23	47Z24
Polymer 1785-47A	100	100	100	100	100	100	100	100	100	100
Maglite D	5*	5*	5*	5*	5*	5*	5*	5*	5*	5*
Dyphos		5*					5*			
Dythal XL			5*					5*		
Leadstar				5*					5*	
SAF Black					1*					1*
Aerosil R972	23*	23*	23*	23*	23*	28*	28*	28*	28*	28*
Mulik 321 SP	40*	40*	40*	40*	40*					
Silane AL72	1	1	1	1	1	1	1	1	1	1
Vulcup 40 KE	5	5	5	5	5	4	4	4	4	4
Press cure, Min./°C	20/160	→	→	→	→	→	→	→	→	→
Oven cure, Hr./°C	1/100	→	→	→	→	→	→	→	→	→
Special Treatment	*Oven dried @ 127°C									
Tensile, Kg/cm ²	85.8	87.9	73.8	47.1	84.4	73.1	77.3	64.0	35.2	80.9
Ult. Elongation, %	100	100	100	100	100	200	200	210	180	200
Shore A Durometer	70	72	68	64	70	62	62	61	59	61
After Aging 70 Hrs. @ 150°C										
Tensile, Kg/cm ²	42.1	45.7	42.2	21.1	40.1	26.7	35.1	24.6	7.7	21.1
Ult. Elongation, %	50	50	50	60	50	110	100	100	80	120
Shore A Durometer	74	76	77	72	76	69	72	70	64	72

TABLE III
Compounds of Base Polymer 1931-07
(Parts by Weight and Test Results)

Batch No.	OLA	OLB	OLC	OLD	OLE	OLF	OLG	OLH	OLI	OLJ	OLK	OLL	OLM	OLN	OLP	OLQ	OLA	OLB	OLC
Polymer 1931-07	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Magnesium oxide	6	6	6							5			5		5		5	5	5
Dyphos		5								5	5								
Zinc oxide			5				3	5	5			5	5			5			5
Calcium hydroxide				6	6	6	6	5			5	5							
Cadmium stearate														6			3		
Zinc stearate																		3	
Stearic acid							1												
Diethyladipate							10												
Aerosil R972			40					20		20	20	25							
Mulok 321 SP								40		40	40	35							
Hsil 233					40	30	30		20										
Mistron vapor									20										
ISAF black													40	40	30	30		30	
MFC black																			30
HAF black																			2
Silane A 172						1		1.5	1.5	1.5	1.5	1.5		1.5					
Carboxax 4000									1.5										
Vulcup 40 KE	6	6	6	6	6	6	6	5	5	5	5	5	5	5	5	5	5	7	7
Press cure, Min./°C	25/160	→	→	→	→	→	→	→	→	→	→	→	20/160	→	→	→	→	→	→
Oven cure, Hr./°C	1/121	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→	→
Special Treatment																			
Tensile, Kg/cm ²	14.1	19.7	10.5	77.3	54.8	49.2	Sponged	98.4	45.7	91.4	91.4	77.3	No	No	14.1	No	24.6	56.2	No
Ult. Elongation, %	200	150	140	120	90	100		100	200	100	100	100	Cure	Cure	200	Cure	400	300	Cure
Shore A Durometer	33	36	30	74	90	60		78	50	72	61	62			40		42	52	

TABLE III, Cont.

Batch No.	O2D	O2E	O2F	O2G	O2H	O2I	O2J	O2K	O7A	O7B	O7BE	O7BF	O7D	O7E	O7F	O7G	O7H	O7I
Polymer 1931-07	100	100	100	50*	100	100	100	100	100	100	50	70	80	100	100	100	100	100
Royalene 400				50*							50	30	20					
Magnesium oxide		5			6	5	5	5	5	5*	5	5	5	5	5	5	5	5
Dyphos						5	5		5	5*	5	5	5	5	5	5	5	5
Cadmium stearate			5															
Zinc stearate				5*														
Calcium hydroxide	4							5										
Agarite Resin D			1.5	1.5*														
Chemlink 30					1.5													
HAF black	30	30	30	30*	40													
Mulok 321 SP						40	40	40	40	40*	40	40	40			20	30	40
Aerosil R972					20	15	20	20		23*	23	23	23	40	40	40	30	20
Aerosil 20C									18									
Silane Al72	2	2				1	1	1.5	1	1	1	1	1					
Carbowax 4000												2	2		2	3	3	3
Vulcup 40 KB	7	7	7	7	6	5	5	5	5	5	5	5	5	5	5	5	5	5
Press cure, Min./°C	20/160	→	→	→	25/160	20/160	→	→	→	→	→	→	→	→	→	25/160	→	→
Oven cure, Hr./°C	2/121	→	1/121	→	→	1/100	→	→	→	→	→	→	→	→	→	→	→	→
Special Treatment				*Bin Aged 2 Wks @ Rm.T.						*Oven dried @ 127°C								
Tensile, Kg/cm ²	No	No	No	63.3	No	84.4	84.4	84.4	73.8	98.4	59.8	36.9	35.2	56.2	84.4	71.7	87.9	73.8
Ult. Elongation, %	Cure	Cure	Cure	650	Cure	110	90	110	70	90	400	160	360	100	200	140	120	120
Shore A Durometer				50		65	70	67	70	62	55	63	60	66	62	70	60	55

TABLE III. Cont.

Batch No.	07J	07K	07L	07M	07N	07O
Polymer 1971-07	100	100	100	100	100	100
Magnesium oxide	5	6	6	6	5	
Dynphos	3				5	5
Cadmium stearate	3					3
Aerosil R972	30		30	30	23	23
Hisil 233		30				
Mistron Vapor				20		
Nulok 321 SP	30				40	40
Silane AL72			1	1	1	1
Carbowax 4000	2	2	2	2		
Vulcup 40 KB	5	4	4	5	5	5
Press cure, Min./ C	25/160	20/160	→	→	→	→
Oven cure, Hr./ C	1/100	1/111	→	→	1/100	→
Tensile, Kg/cm ²	42.2	42.2	45.1	49.2	91.4	73.8
Ult. Elongation, %	220	220	300	130	80	140
Shore A Durometer	53	57	59	85	64	62

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